

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE
REQUEST FOR FILING NATIONAL PHASE OF
PCT APPLICATION UNDER 35 U.S.C. 371 AND 37 CFR 1.494 OR 1.495

To: Hon. Commissioner of Patents
Washington, D.C. 20231

00909

TRANSMITTAL LETTER TO THE UNITED STATES
DESIGNATED/ELECTED OFFICE (DO/EO/US)

Atty Dkt: P 291086 /M80283871:DVG:cl
M# /Client Ref.

From: Pillsbury Winthrop LLP, IP Group:

Date: March 15, 2002

This is a **REQUEST** for **FILING** a PCT/USA National Phase Application based on:

1. International Application	2. International Filing Date	3. Earliest Priority Date Claimed
<u>PCT/AU00/01131</u>	18 September 2000	17 September 1999
<u> ↑ country code</u>	Day MONTH Year	Day MONTH Year

4. Measured from the earliest priority date in item 3, this PCT/USA National Phase Application Request is being filed within:

(a) 20 months from above item 3 date (b) 30-months from above item 3 date,
(c) Therefore, the due date (unextendable) is March 17, 2002

5. Title of Invention PROCESS FOR PREPARING FOOD CONTACT GRADE POLYETHYLENE TEREPHTHALATE RESIN FROM WASTE PET CONTAINERS

6. Inventor(s) KOSIOR, Edward

Applicant herewith submits the following under 35 U.S.C. 371 to effect filing:

7. Please immediately start national examination procedures (35 U.S.C. 371 (f)).
8. **A copy of the International Application** as filed (35 U.S.C. 371(c)(2)) is transmitted herewith (file if in English but, if in foreign language, file only if not transmitted to PTO by the International Bureau) including:
 - a. Request;
 - b. Abstract;
 - c. 11 pgs. Spec. and Claims;
 - d. sheet(s) Drawing which are informal formal of size A4 11"
9. **A copy of the International Application has been transmitted by the International Bureau.**
10. **A translation of the International Application** into English (35 U.S.C. 371(c)(2))
 - a. is transmitted herewith including: (1) Request; (2) Abstract; (3) pgs. Spec. and Claims; (4) sheet(s) Drawing which are: informal formal of size A4 11"
 - b. is not required, as the application was filed in English.
 - c. is not herewith, but will be filed when required by the forthcoming PTO Missing Requirements Notice per Rule 494(c) if box 4(a) is X'd or Rule 495(c) if box 4(b) is X'd.
 - d. Translation verification attached (not required now).

11. Please see the attached Preliminary Amendment

12. Amendments to the claims of the International Application **under PCT Article 19 (35 U.S.C. 371(c)(3))**, i.e., before 18th month from first priority date above in item 3, are transmitted herewith (file only if in English) including:

13. PCT Article 19 claim amendments (if any) have been transmitted by the International Bureau

14. Translation of the amendments to the claims **under PCT Article 19 (35 U.S.C. 371(c)(3))**, i.e., of **claim amendments** made before 18th month, **is attached (required by 20th month from the date in item 3 if box 4(a) above is X'd, or 30th month if box 4(b) is X'd, or else amendments will be considered canceled)**.

15. **A declaration of the inventor (35 U.S.C. 371(c)(4))**

a. is submitted herewith Original Facsimile/Copy

b. is not herewith, but will be filed when required by the forthcoming PTO Missing Requirements Notice per Rule 494(c) if box 4(a) is X'd or Rule 495(c) if box 4(b) is X'd.

16. **An International Search Report (ISR):**

a. Was prepared by European Patent Office Japanese Patent Office Other

b. has been transmitted by the international Bureau to PTO.

c. copy herewith (2 pg(s).) plus Annex of family members (1 pg(s).)

17. **International Preliminary Examination Report (IPER):**

a. has been transmitted (if this letter is filed after 28 months from date in item 3) in English by the International Bureau with Annexes (if any) in original language.

b. copy herewith in English.

c. 1 IPER Annex(es) in original language ("Annexes" are amendments made to claims/spec/drawings during Examination) including attached amended:

c. 2 Specification/claim pages #1 - 13 claims #1 - 11
Dwg Sheets #

d. Translation of Annex(es) to IPER **(required by 30th month due date, or else annexed amendments will be considered canceled)**.

18. **Information Disclosure Statement** including:

a. Attached Form PTO-1449 listing documents

b. Attached copies of documents listed on Form PTO-1449

c. A concise explanation of relevance of ISR references is given in the ISR.

19. **Assignment** document and Cover Sheet for recording are attached. Please mail the recorded assignment document back to the person whose signature, name and address appear at the end of this letter.

20. Copy of Power to IA agent.

21. **Drawings** (complete only if 8d or 10a(4) not completed): ___ sheet(s) per set: 1 set informal; Formal of size A4 11"

22. Small Entity Status is **Not** claimed is claimed (**pre-filing confirmation required**)
22(a) _____ (No.) Small Entity Statement(s) enclosed (since 9/8/00 Small Entity Statements(s) not essential to make claim)

23. **Priority** is hereby claimed under 35 U.S.C. 119/365 based on the priority claim and the certified copy, both filed in the International Application during the international stage based on the filing in (country) **AUSTRALIA** of:

	Application No.	Filing Date	Application No.	Filing Date
(1)	PQ 2946	Sept. 17, 1999	(2)	
(3)			(4)	
(5)			(6)	

a. See Form PCT/IB/304 sent to US/DO with copy of priority documents. If copy has not been received, please proceed promptly to obtain same from the IB.

b. Copy of Form PCT/IB/304 attached.

24. Attached:

25. Per Item 17.c2, cancel original pages # _____, claims # _____, Drawing Sheets # _____26. **Calculation of the U.S. National Fee (35 U.S.C. 371 (c)(1)) and other fees is as follows:**Based on amended claim(s) per above item(s) 12, 14, 17, 25 (hilite)

Total Effective Claims	minus 20 =	$\times \$18/\9	= \$0	966/967
Independent Claims	minus 3 =	$\times \$84/\42	= \$0	964/965
If any proper (ignore improper) Multiple Dependent claim is present,		add\$280/\$140	+0	968/969

BASIC NATIONAL FEE (37 CFR 1.492(a)(1)-(4)): →→ BASIC FEE REQUIRED, NOW →→→→

A. If country code letters in item 1 are not "US", "BR", "BB", "TT", "MX", "IL", "NZ", "IN", "ZA", "LC" or "PH"

See item 16 re:

1. Search Report was not prepared by EPO or JPO -----	add\$1,040/\$520	960/961	
2. Search Report was prepared by EPO or JPO -----	add\$890/\$445	+1040	970/971

SKIP B, C, D AND E UNLESS country code letters in item 1 are "US", "BR", "BB", "TT", "MX", "IL", "NZ", "IN", "ZA", "LC" or "PH"

→ <input type="checkbox"/> B. If USPTO did not issue both International Search Report (ISR) and (if box 4(b) above is X'd) the International Examination Report (IPER), -----	add\$1,040/\$520	+0	960/961
(X) (only) (one) → <input type="checkbox"/> C. If USPTO issued ISR but not IPER (or box 4(a) above is X'd), -----	add\$740/\$370	+0	958/959
(these) (4) → <input type="checkbox"/> D. If USPTO issued IPER but IPER Sec. V boxes <u>not all</u> 3 YES, -----	add\$710/\$355	+0	956/957
→ <input type="checkbox"/> E. If international preliminary examination fee was paid to USPTO and Rules 492(a)(4) and 496(b) satisfied (i.e., in IPER Sec. V <u>all</u> 3 boxes <u>must</u> be YES for <u>all</u> claims), -----	add \$100/\$50	+0	962/963

27. **SUBTOTAL = \$1040**28. If Assignment box 19 above is X'd, add Assignment Recording fee of ----\$40 **+0** (581)29. If box 15a is x'd, determine whether inventorship on Declaration is different than in international stage. If yes, add (per Rule 497(d)) **----\$130** **+0** (098)30. Attached is a check to cover the ----- **TOTAL FEES \$1040**

Our Deposit Account No. 03-3975

Our Order No. (7287) (291086)
C# _____ M# _____

00909

CHARGE STATEMENT: The Commissioner is hereby authorized to charge any fee specifically authorized hereafter, or any missing or insufficient fee(s) filed, or asserted to be filed, or which should have been filed herewith or concerning any paper filed hereafter, and which may be required under Rules 16-18 and 492 (missing or insufficient fee only) now or hereafter relative to this application and the resulting Official document under Rule 20, or credit any overpayment, to our Account/Order Nos. shown above for which purpose a duplicate copy of this sheet is attached.

This CHARGE STATEMENT does not authorize charge of the issue fee until/unless an issue fee transmittal form is filed

Pillsbury Winthrop LLP
Intellectual Property Group

By Atty: Glenn J. PerryReg. No. 28458

Sig:

Fax: (703) 905-2500
Tel: (703) 905-2161

Atty/Sec: gjp/mhn

NOTE: File in duplicate with 2 postcard receipts (PAT-103) & attachments.

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re PATENT APPLICATION OF

Inventor(s): KOSIOR, Edward

Filed: Herewith

Title: PROCESS FOR PREPARING FOOD CONTACT GRADE POLYETHYLENE
TEREPHTHALATE RESIN FROM WASTE PET CONTAINERS

March 15, 2002

PRELIMINARY AMENDMENT

Hon. Commissioner of Patents
Washington, D.C. 20231

Sir:

Please amend this application as follows:

IN THE SPECIFICATION:

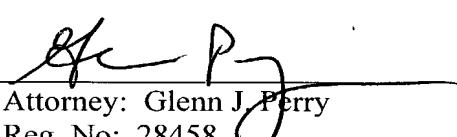
At the top of the first page, just under the title, insert

--This application is the National Phase of International Application
PCT/AU00/01131 filed Sept. 18, 2000 which designated the U.S.
and that International Application
 was was not published under PCT Article 21(2) in English.--

Respectfully submitted,

PILLSBURY WINTHROP LLP
Intellectual Property Group

By:


Attorney: Glenn J. Perry
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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

COMPLETION OF FILING NATIONAL PHASE OF PCT APPLICATION
UNDER RULE 35 USC 371 AND 37 CFR 1.494(C) OR 1.495(C)

BOX PCT

**COMPLETION
For PCT Cases Only**In re PATENT APPLICATION of
Inventor(s): Edward KOSIORAppln. No.: 10 Atty. Dkt. P 291086 M80283871:DVG/cl
Series Code ↑ Serial No. ↑ M# Client Ref

National Phase

Based on PCT AU00, 01131
↑ Country Code & Year

Attn: Application Division

Title: Process for Preparing Food Contact Grade Polyethylene
Terephthalate Resin From Waste Pet Containers

Date: June 24, 2002

FILING OF ITEM(S) LATE IN PCT/USA NATIONAL CASEAsst. Commissioner of Patents
Washington, DC 20231

Sir:

The following completes the filing of the subject application under Rule 494(c)/495(c). Please accept the following attached items:

1. Missing Requirements Notice (PCT/DO/EO/905) copy attached not yet received
2. **Signed Declaration** Original Facsimile/Copy with spec/claims attached
3. **Translation** of the International Application into English including:
 - a. Request;
 - b. Abstract
 - c. pgs. Spec. and Claims;
 - d. Translation verification formal of size A4 11"
4. Copy of **International Search Report** (ISR) attached (____ page(s))
 - a. plus Annex of family members (____ page(s))
5. **Information Disclosure Statement** including
 - a. From PTO-1449 listing documents
 - b. Copies of document(s) listed on Form PTO-1449
 - c. A concise explanation of ISR references is given in the ISR
6. **Assignment** and cover sheet. Please return the recorded assignment to the undersigned.
7. Copy of Power to international application agent.
8. ____ (No.) Small Entity Statement(s) establishing "small entity" status under Rules 9 & 27.
9. Formal Drawings: ____ sheet(s) informal; formal of size: A4 11"
10. Please immediately start national examination procedures (35 USC 371(f))

11. Attached:
 12. Preliminary Amendment:
 13. Basic U.S. National fee per Rule 492(a)(1)-(4) was previously timely filed.
 14. **Calculation of remaining fees due (if any):** based on amended claim(s) per above item
 12 (above) or item(s) in PAT-112 (filed previously) 12 14 17 25

15. **CLAIMS FEES** previously paid paid herewith as follows:

15A. Small Entity Statement Herewith Previously Filed

				Large/Small Entity		Fee Code
16. Total Effective Claims	12	minus 20 =	0	x \$18/\$9	+0	966/967
17. Independent Claims	1	minus 3 =	0	x \$84/\$42	+0	964/965
18. If <u>any</u> proper multiple dependent claim (ignore improper) is present,				\$280/\$140	+280	968/969
19. Filing Declaration late, fee paid	<input type="checkbox"/> previously	<input checked="" type="checkbox"/> now		\$130/\$65	+130	154/254
20.				SUBTOTAL	\$410	
21. Original due date:	July 14, 2002					
22. Petition is hereby made to extend the <u>original</u> due date to	(1 mo)	\$110/\$55 =	+0	115/215		
cover the date this response is filed for which the requisite fee	(2mos)	\$400/\$200 =		116/216		
is attached	(3mos)	\$920/\$460 =		117/217		
	(4mos)	\$1,440/\$720 =		118/218		
23. If "non-English" box 3 is X'd, add Rule 17(k) processing fee	\$130	+0		156		
24. If "assignment" box 6 is X'd, add recording fee.	\$40	+40		581		
25.		TOTAL FEE =	\$450			
				PLEASE CHARGE	OUR DEP. ACCT.	
(Our Deposit Account No. 03-3975)						
(Our Order No. 7287		291086				
	C#	M#				

CHARGE STATEMENT: The Commissioner is hereby authorized to charge any fee specifically authorized hereafter, or any missing or insufficient fee(s) filed, or asserted to be filed, or which should have been filed herewith or concerning any paper filed hereafter, and which may be required under Rules 16-18 (missing or insufficient fee only) now or hereafter relative to this application and the resulting Official document under Rule 20, or credit any overpayment, to our Account/Order Nos. shown above for which purpose a duplicate copy of this sheet is attached.

This CHARGE STATEMENT does not authorize charge of the issue fee until/unless an issue fee transmittal form is filed.

Pillsbury Winthrop LLP
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NOTE: File in duplicate with PTO receipt (PAT-103A) and attachments

08/09/2002 CBURT1 00000001 033975 10008204

01 FC:968 280.00 CH

PROCESS FOR PREPARING FOOD CONTACT GRADE POLYETHYLENE TEREPHTHALATE RESIN FROM WASTE PET CONTAINERS

Technical Field

5 This invention relates to methods for preparing polyethylene terephthalate (PET) resin from plastic waste that includes PET containers. It also relates to PET obtained according to the process.

Background to the Invention

PET is a widely used polymer or resin with a broad range of applications but which has a particularly widespread use as a beverage container or bottle. The use of PET for beverage containers has increased rapidly over the last decade and has to a large extent replaced conventional glass beverage containers especially for carbonated soft drinks. Part of the widespread acceptance of PET has been attributed to its ability to be used for food contact as well as its light weight relative to glass of comparable strength and its ability to resist breakage.

Over recent years environmental pressures have increased and there is a demand for the recycling of many materials, especially plastics. One common source of recyclable material is post consumer curbside waste. With PET containers most of the applications for recycled PET are for relatively low specification products that use a mixture of thermoplastic resins or polymers including PET. In these applications removal of contaminants is not important. It is desirable that PET containers may be recycled to produce PET resin that is suitable for the same applications as virgin PET. For example, it is especially desirable that the recycled resin may be used for food contact applications. However, for such applications there are strict limits on the presence of contaminants.

Various methods have been proposed for recycling PET resin. One such method is disclosed in US Patent No. 5554657 which is assigned to the Shell Oil Company. In this patent a mixed polymer recycle stream that includes PET polymers is contacted with a solvent that selectively dissolves PET. This

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- 2 -

polymer solution is then separated from undissolved material and cooled to allow the precipitation of the PET resin. While this process leads to excellent quality product it is expensive as it involves the use of organic solvents which need to be recycled themselves. Other recycling methods involve the separation of particulate contaminants by filtration of a molten polymer. This filtration method is facilitated by reducing the molecular weight and thus the intrinsic viscosity of the polymer to allow the molten material to flow more readily. However this reduction in molecular weight necessitates a final polymerisation or condensation reaction stage to produce PET of the required viscosity and molecular weight. This method also requires the frequent replacement and/or cleaning of filters.

A further approach to recycling PET for food grade applications is disclosed in Australian Patent Application No. 9478299. In this invention beverage containers are produced having inner and outer skins. The inner skin which is in contact with food is made from virgin PET and the outer skin is made from recycled PET resin. This invention removes the necessity for the recycled PET being suitable for food contact. However it is desirable that recycled PET can be used for direct food applications without using multiple skin production methods with their associated complexities and costs.

US Patent 5,876,644 discloses a process for preparing food contact grade PET. The process involves the surface cleaning of comminuted pieces of post consumer PET containers; followed by melting of the cleaned pieces; followed by extrusion to form a melt and then blending of this melt with a melt virgin polyester prepolymer. The combined melt is then solidified and polymerisation is then effected while the pellets are in the solid state. The use of virgin PET prepolymer would have the effect of reducing the contaminant level as well as allowing solid state polymerisation to take place to achieve the desired intrinsic viscosity increase. It is an important feature of this earlier invention that molecular weight increase takes place in the solid state after extrusion. As this process requires a post extrusion solid state increase in intrinsic viscosity a

AMENDED SHEET

Received 25 October 2001

- 3 -

reduction in the intrinsic viscosity during extrusion is permitted or achieved by adding water prior to melting and extrusion (see column 7, lines 25-35).

PET originating from sorted collections of solid urban refuse can be contaminated with a range of materials. The PET will include both food contact 5 grade PET such as containers as well as non food contact grade PET. The range of non PET materials include other polymers especially polyolefins such as HDPE. Other common contaminants are metals, particulate material such as dirt, glues, paper, inks and remnants of materials stored in the containers. It is desirable that food contact grade PET may be obtained from such collections of 10 urban refuse.

Summary of the Invention

This invention provides in one form a process for preparing food contact grade PET from a waste stream containing PET and non PET materials comprising the following steps:

- 15 sorting at least some of the non PET materials from the waste stream;
- dividing the PET containers into flakes of preferable maximum size approximately 10mm;
- washing the flakes in a hot aqueous medium containing alkaline materials and surfactants, preferably non-ionic, to remove particulate and 20 absorbed contaminants from the surfaces of the flakes;
- de-watering and then drying the flakes to a moisture content of 0.1% w/w maximum, and more preferably 0.01% w/w maximum;
- optionally removing absorbed contaminants and moisture by heating and vigorously mixing the flakes under vacuum, preferably 1- 10 millibar, more 25 preferably 2 – 7 millibar and at a temperature less than the melting point of PET, preferably in the range 170-220°C for at least 30 minutes, preferably at least 60 minutes;
- melting the flakes in a screw extruder under vacuum to remove absorbed contaminant and;
- 30 extruding the molten material to form strands that are pelletised.

AMENDED SHEET

- 4 -

Preferably the material in the extruder is maintained at 280 - 290°C with a residence time of less than 60 seconds.

Preferably the heating to remove the absorbed contaminants is attained by frictional forces from the vigorous mixing.

5 Detailed Description of the Invention

To meet FDA requirements for food contact grade PET the recycled PET must have contamination sufficiently low than such that the level of extractables is less than 10 ppb. As well as contaminants from the presence of non PET containers such as dirt, and other plastics, a range of materials may be adsorbed into or absorbed onto PET surfaces. These contaminants can include organo metallic materials such as copper octoate. Adsorbed materials may include polar and non polar organic materials that have a range of volatilities.

It is an important feature of the present invention that the majority of adsorbed and absorbed materials are removed while the PET material is in flake form. We have found this facilitates the removal of contaminants that are generally either on or near the surface of the PET flakes. We have found that the removal of such contaminants after the material has become molten is much less effective as the contaminants tend to become buried in the resinous mass. It is also an important feature of the present invention that water content is reduced to low levels and is further reduced during the vacuum venting of the PET melt in the screw extruder. We have found the low initial water level and the vacuum venting in the melt extruder enables the equilibrium water content to be reduced such that the molecular weight is increased. The presence of excess levels of water at melt temperatures tends to cause hydrolysis of the ester linkage leading to reduced molecular weight and thus the intrinsic viscosity of the resin.

This invention will be further described by reference to preferred processes.

An urban solid waste stream consisting of baled bottles are passed through a debaler that singulates the bottles so that they can be fed at a steady

AMENDED SHEET

15/03/2002

- 5 -

rate to the inlet of a pre-wash unit. The preferred pre-wash unit is one that utilises elevated temperatures and alkaline surfactants such as one like a Sorema Bottle Pre-wash unit. However similar units that have either a continuous or batch wise mode of operation may also be used.

5 The feed rate is typically in the range 500 kgs to 2500 kgs per hour with 1500 kgs per hour being the optimum rate. The action of the pre-wash unit is to tumble the bottles using the rotary motion of the cylindrical tumbler unit about its longitudinal axis. Internal baffles in the tumbler ensure that all bottles are singulated by repeated impact of the bottles falling against the walls and baffles
10 while they are exposed to hot water and steam. The internal temperature is typically maintained in the range 90 to 100°C with temperatures greater than 95°C being preferred. The residence time in the pre-wash unit is generally in the range between 3 minutes and 15 minutes with a typical time being 5 minutes. The water in the pre-wash may include cleaning agents such as caustic
15 soda and non-foaming detergents. Typical concentrations of the caustic soda and detergent are 0.1 to 3% (ideally 0.5%) and 0.1 to 0.5% (ideally 0.2%) respectively.

The preferred detergent or surfactant is non-ionic.

20 The wash bottles are then de-watered by tumbling them in a cylindrical tumbler or similar device that allows the freed dirt and other contaminants such as labels and closures to pass through the perforations in the walls of the tumbler. The water can be reused after it is filtered and treated to remove foreign materials. The residence time in this de-watering unit is in the range 3 minutes and 15 minutes with a typical time being 5 minutes.

25 At this stage the bottles are clean externally except for a film of water and are mostly free of plastic or paper labels through the action of the mechanical handling of the bottles, the hot water and the cleaning agents.

The PET bottles are then sorted. The preferred process uses automatic systems such as those made by Magnetic Separation Systems (MSS), ROFIN or
30 National Recovery Technologies Inc (NRT) although manual sorting can also be

AMENDED SHEET

- 6 -

used. Particularly good results are achieved when a sequence of compositional analysis and sorting modules are used to sort the PET bottles to give a level of purity of no more than 20 ppm of PVC. The level of sorting of non PET should be to 99.998% purity. The MSS modules use modular sensors to detect the presence of specific plastics and air jets to eject the bottles at a specific station.

The first module uses an X-ray absorption to detect the chlorine atom in PVC and the PVC bottles are ejected at this station. This module also removes aluminium cans due to their strong X-ray absorption.

The second module uses infra-red absorption to detect HDPE (high density polyethylene) bottles and these are then ejected.

The third module uses near infra-red absorption to detect PET and in this module all the non PET bottles are ejected. This module will eject bottles such as PVC, HDPE, polypropylene, polystyrene and aluminium cans.

The fourth module uses X-ray absorption to detect the chlorine atom in PVC and the PVC bottles are ejected at this station. This module also removes aluminium cans due to their strong X-ray absorption.

A manual inspection is used to finally check that only PET bottles proceed into the later stages of the process.

The sorted PET containers are then reduced in size using a wet grinder such as a Sorema hot wash, separation and rinsing system or its equivalent.

The wet grinder uses multiple rotating knives to cut the PET bottles against stationary knives in the presence of water that is at ambient temperature or at elevated temperatures (from 10 to 40°C, with 15 to 20°C being most often used), and which will contain caustic soda and low foaming surfactants and antifoam additives. Typical concentrations of the caustic soda and detergent is 0.1 to 3% (ideally 0.5%) and 0.1 to 0.5% (ideally 0.2%) respectively. Antifoam use is related to surfactant level and is usually in the range 0.01 to 1%.

The PET bottles are cut against a screen with a hole size of 10mm to 30mm with 16 to 20 mm being the most common. This gives an intense washing and simultaneous cutting effect on the PET bottles resulting in a range

AMENDED SHEET

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Received 25 October 2001

- 7 -

of average flake sizes that varies from 3mm to 12mm with the most numerous being under 8mm.

After grinding to this small size the mixture of PET flake and polypropylene particles (from closures and neck rings) is fed into a hot wash station such as Sorema Hot Wash Reactors (or similar) where the mix is intensively washed for 10 to 20 minutes at temperatures from 75 to 95°C with 90°C being the ideal. The flakes are fed into the reactors at a liquid to plastic flake slurry ratio of 90/10 to 65/35 with 75/25 Volume/Volume being most common.

10 The reactors are designed to provide turbulent washing conditions where particles impinge on each other through the use of opposed-rotor, dual-rotor stirrers that are used in non-central positions to prevent laminar mixing from occurring, thus ensuring the most intense washing effect on the PET flakes.

After washing the flakes are separated from the wash solution by the use of a centrifuge or screen, and the flakes are subjected to a sink-float separation in a tank of water where the polypropylene particles float due to their density being less than that of water (915 kg/m^3) and the PET particles sink due to their density being greater than that of water (1400 kg/m^3).

The separated PET flakes are then further rinsed at least twice in clean water to remove the residual traces of surfactants and dilute contaminants. The pathway of the water and flakes is counter current to provide the maximum rising effect.

The PET flakes are then de-watered to give a very low level of moisture, i.e. down to 0.005% water.

25 This can be done by staged drying with fluidised bed driers to remove apparent moisture followed by conventional recirculating air driers, desiccant driers, agglomerators or other drying systems that may also use a dry gas to dry the PET at elevated temperatures (140 – 185°C).

The fluidised bed driers will remove the moisture from saturated levels down to levels of less than 1% and typically 0.5%.

AMENDED SHEET

PC/PA000/01131
Received 25 October 2001

- 8 -

The desiccant driers, agglomerators or other driers will reduce the moisture level to at least 0.01% i.e. 1000ppm of water with an ideal level of 0.005% of moisture, i.e. 500ppm. In conventional driers this may require the use of long residence times as well as high temperatures, e.g. temperatures of the order of 120 to 175°C for 8 to 5 hours with 170°C for 5 hours proving optimum. The use of pre drying of the PET flakes is important as it leads to an increase rather than decrease in the intrinsic viscosity (IV) of the polymer in the following stages of the process. For example, the IV of the final PET pellet with the pre drying step was 0.833 versus 0.749 without compared to the IV of the flake itself of 0.767.

The dried flakes are then subjected to vacuum decontamination.

This process uses high levels of vacuum, preferably 1- 10 millibar, more preferably 2 – 7 millibar, while the PET is subjected to elevated temperatures (170 to 215°C) and mixing for controlled residence times of typically 1 hour, 15 although longer times may also be useful. This decontamination can be performed in a shredder chamber modified to maintain a vacuum, or in a fluidising mixer modified to hold a vacuum. Frictional forces between flakes and parts of the equipment lead to heat build up and this is the preferred method of attaining the desired temperature. The impeller in the chamber is rotated at 20 between 200 to 220 rpm converting mechanical energy into heat. Increasing the speed generates high temperatures with typically a temperature gradient, the higher temperatures being at the bottom. For example, when the impeller speed was 220 rpm the temperature at the bottom was 199°C, in the middle was 189°C and at the top 169°C.

25 Preferably the loading of the chamber with the PET flakes and the rotor speed are selected so that the chamber was filled to a sufficiently high level, approximately 70%, that flake introduced through a vacuum lock could reside at the top for a controlled residence time without the risk of immediately being mixed into the bulk of the flake and being extruded with only a short residence time. The conditions in the chamber were balanced so that the PET flake was

AMENDED SHEET

Received 25 October 2001

- 9 -

progressively exposed to higher temperatures while under high vacuum and maintained at this condition for at least one hour after which it was introduced into the extruder.

5 The decontamination of the PET takes place in the mixer by the combined action of the vacuum, elevated temperature and the residence time of the PET under these conditions.

The decontaminated PET flakes are then fed to a single screw extruder with an extrusion screw designed for the processing of PET and capable of applying vacuum venting to the PET melt at 280 to 290°C. The process could 10 be conducted in a similar twin screw extruder with vacuum venting or in a twin or multi-screw extruder with vacuum venting. The key requirement is the capability to melt the PET (melt temperature in the range 265 – 300°C, preferably 280°C) and to apply one or more stages of vacuum venting (at preferably 1 millibar or less) without applying excessive shear to the PET in the 15 melt stage through excessive mechanical working of the melt. Excessive shear leads to a decrease in the IV. The application of the higher temperatures in the melt coupled with the vacuum venting allows removal of the least volatile fluids that may have been absorbed into the PET.

While this process has been described without the use of chemical chain 20 extenders to increase the IV of the PET, these chemical materials may also be used. Chain extender materials are known and usually comprise one or more of a polycarboxylic acid or anhydride, a polyol and an esterification catalyst. For example, we have found a mixture of pyromellitic dianhydride, anhydride, pentaerythritol and antimony oxide in the weight ratios of 4:1:0.5 is a 25 particularly useful chain extender composition. In the process described above the use of this chain extender composition has been able to increase the IV of the PET to 0.930 when used at 0.3% w/w of PET. Higher levels, eg 1.0% w/w increased the IV to 1.300.

AMENDED SHEET

Received 25 October 2001

10

After melting and vacuum venting the melt is filtered by passing it through fine metal mesh filters (usually the mesh size is 120 mesh or finer) to remove any particles.

The extruded melt may then be converted to pellets typically 3mm using conventional techniques such as by using an underwater die face cutter or hot die face and water ring that quenches the melt into pellets as they are cut. Further steps may include crystallising the pellets and pre-drying the pellets prior to moulding. The crystallising is carried out by heating the PET pellets whilst they are kept moving via tumbling or agitation. The temperature is maintained at from 120°C to 170°C for between 10 minutes and 1 hour.

Pre drying is carried out by heating the pellets at elevated temperatures in hot gas that has a dew point of less than 40°C. The temperatures used typically vary from 140°C to 190°C for a duration that typically varies from 4 hours to 7 hours.

The effectiveness of the recycling process of the present invention is illustrated by introducing the following contaminants to PET containers:

toluene 10% v/v, chloroform 10% v/v, benzophenone 1% v/v, methyl stearate 1% v/v and copper octoate 1% v/v.

The concentrations of these contaminants after the various stages of the process steps of the present invention are set out in Table 1. These results show that after the process of the present invention contamination levels in the extruded pellets are acceptably low. The Table also shows the levels of extractables from PET bottles made from pellets prepared as described above.

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PCT/AU00/01131
Received 25 October 2001

- 11 -

Table 1
**Contaminant Levels (Ppm) In Washed Flake,
Extruded Pellets And Food Stimulant (10% Ethanol)**

Contaminant	PET	PET Flake	Extruded	Levels in Food
	Flake Before Wash	After Wash	PET Pellets	Simulant (10% Ethanol) After the Migration test with Bottles made from PET Pellets
Toluene	1768.0	360.5	7.0	<0.01
Chloroform	612.5	52.3	24.8	<0.004
Benzophenone	713.3	175	54	<0.005
methyl stearate	81.2	16	1	<0.005
copper octoate	230.3	8	5.0	<0.001

Received 25 October 2001

- 12 -

CLAIMS:

1. A process for preparing food contact grade PET from a waste stream containing PET and non PET materials comprising the following steps:
 - sorting at least some of the non PET materials from the waste stream;
 - 5 dividing the PET containers into flakes;
 - washing the flakes in a hot aqueous medium containing alkaline materials and surfactants to remove particulate and absorbed contaminants from the surfaces of the flakes;
 - 10 de-watering and then drying the flakes to a moisture content of 0.1% w/w maximum;
 - melting the flakes in a screw extruder under vacuum to remove absorbed contaminants; and
 - extruding the molten material to form strands that are pelletised.
- 15 2. A process according to claim 1 wherein there is a further heating and mixing step, before the extruder, that heats and vigorously mixes the flakes under vacuum at a temperature less than the melting point of PET.
- 20 3. A process according to claim 1 or claim 2 wherein the surfactants in the flake washing step are non-ionic.
4. A process according to any one of claims 1 – 3 wherein the maximum moisture content of the flakes after the drying step is 0.01% w/w.
- 25 5. A process according to any one of claims 2 – 4 wherein the heating and mixing step is conducted at a reduced pressure of 1 – 10 millibar.
6. A process according to claim 5 wherein the pressure is in the range of 2 – 7 millibar.

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Received 25 October 2001

- 13 -

7. A process according to any one of claims 2 – 6 wherein the heating and mixing step is conducted at a temperature in the range 170 – 200°C for at least 30 minutes.
- 5 8. A process according to claim 7 wherein the heating and mixing step is conducted for at least 60 minutes.
9. A process according to any one of claims 1 – 8 wherein the material in the extruder is maintained at a temperature in the range 280 – 290°C for less than sixty seconds at a reduced pressure of 1 millibar or less.
- 10 10. A process according to any one of claims 1 – 9 wherein a chemical chain extender is used to increase the molecular weight of the recycled PET.
- 15 11. Recycled food contact grade PET prepared according to a process as defined in any one of claims 1 – 10.

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A1

(54) Title: **PROCESS FOR PREPARING FOOD CONTACT GRADE POLYETHYLENE TEREPHTHALATE RESIN FROM WASTE PET CONTAINERS**

WO 01/21373

(57) Abstract: A process for preparing food contact grade PET from a waste stream containing PET and non PET materials is disclosed. The process involves the steps of sorting at least some of the non PET materials from the waste stream, followed by dividing the PET containers into flakes of preferable maximum size approximately 10mm, followed by washing the flakes in a hot aqueous medium containing alkaline materials and surfactants, preferably non-ionic, to remove particulate and absorbed contaminants from the surfaces of the flakes. This step is followed by de-watering and then drying the flakes to a moisture content of 0.1 % w/w maximum, and more preferably 0.01 % w/w maximum. The next step involves removing absorbed contaminants and moisture by heating and vigorously mixing the flakes under vacuum, preferably 1-10 millibar, more preferably 2-7 millibar and at a temperature less than the melting point of PET, preferably in the range 170-220 °C for at least 30 minutes, preferably at least 60 minutes. The final step involves melting the flakes in a screw extruder equipped for vacuum processing and extruding the molten material to form strands that are pelletised.

FOR UTILITY/DESIGN
CIP/PCT NATIONAL/PLANT
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DECLARATIONS

3.00088304 - 06-24-02
DECLARATION AND POWER OF ATTORNEY
FOR PATENT APPLICATION
IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

FORM

As a below named inventor, I hereby declare that my residence, post office address and citizenship are as stated below next to my name, and I believe I am the original, first and sole Inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the INVENTION ENTITLED PROCESS FOR PREPARING FOOD CONTACT GRADE POLYETHYLENE TEREPHTHALATE RESIN FROM WASTE PET CONTAINERS

the specification of which CHECK applicable BOX(ES)
 A. is attached hereto.
 BOX(ES) → B. was filed on March 15, 2002 as U.S. Application No. 10/088,204
 → C. was filed as PCT International Application No. PCT/ AU00/01131 on September 18, 2000
 and (if applicable to U.S. or PCT application) was amended on
 I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above. I acknowledge the duty to disclose all information known to me to be material to patentability as defined in 37 C.F.R. 1.56. Except as noted below, I hereby claim priority benefits under 35 U.S.C. 119(a)-(d) or 365(b) of any foreign application(s) for patent or inventor's certificate, or 365(a) of any PCT International Application which designated at least one other country than the United States, listed below and have also identified below any foreign application for patent or inventor's certificate, or PCT International Application, filed by me or my assignee disclosing the subject matter claimed in this application and having a filing date (1) before that of the application on which priority is claimed, or (2) if no priority claimed, before the filing date of this application:

PRIOR FOREIGN APPLICATION(S)		Date first Laid-open or Published	Date Patented or Granted	Priority NOT Claimed
Number	Country			
PQ2946	AUSTRALIA	17 September 1999		

If more prior foreign applications, X box at bottom and continue on attached page.

Except as noted below, I hereby claim domestic priority benefit under 35 U.S.C. 119(e) or 120 and/or 365(c) of the indicated United States applications listed below and PCT international applications listed above or below and, if this is a continuation-in-part (CIP) application, insofar as the subject matter disclosed and claimed in this application is in addition to that disclosed in such prior applications, I acknowledge the duty to disclose all information known to me to be material to patentability as defined in 37 C.F.R. 1.56 which became available between the filing date of each such prior application and the national or PCT international filing date of this application:

PRIOR U.S. PROVISIONAL, NONPROVISIONAL AND/OR PCT APPLICATION(S)	Status	Priority NOT Claimed
Application No. (series code/serial no.)	Day/Month/Year Filed	
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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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24-4-02

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FOR ADDITIONAL INVENTORS see attached page.

See additional foreign priorities on attached page (incorporated herein by reference).

Atty. Dkt. No. P291086

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